

**NASA
Technical
Memorandum**

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**MATERIALS AND PROCESSES LABORATORY
COMPOSITE MATERIALS CHARACTERIZATION TASK
PART I. DAMAGE TOLERANCE**

By A.T. Nettles, D.S. Tucker, W.J. Patterson, S.W. Franklin,
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Materials and Processes Laboratory
Science and Engineering Directorate

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13. ABSTRACT (Maximum 200 words) In an effort to best utilize all areas of expertise within the Materials and Processes Laboratory, a Composite Materials Characterization Task Team was developed to help bring together the various branches within the Laboratory to develop a comprehensive data base on composite materials. A "test run" was performed on IM6/3501-6 carbon/epoxy in which the material was processed, machined into specimens, and tested for damage tolerance capabilities. Nondestructive test data played a major role in this element of composite characterization. A time chart was produced showing the time the composite material spent within each Branch or Division in order to identify those areas which produce a long turnaround time. Instrumented drop weight testing was performed on the specimens with nondestructive evaluation (NDE) being performed before and after the impacts. Destructive testing in the form of cross-sectional photomicrography and compression-after-impact (CAI) testing were used. Results show that the processing and machining steps needed to be performed more rapidly if data on a composite material is to be collected within a reasonable timeframe. The results of the damage tolerance testing showed that IM6/3501-6 is a brittle material that is very susceptible to impact damage.					
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TECHNICAL MEMORANDUM

MATERIALS AND PROCESSES LABORATORY COMPOSITE MATERIALS CHARACTERIZATION TASK, PART I. DAMAGE TOLERANCE

I. INTRODUCTION

With the advent of stronger, lighter composite materials, it is becoming increasingly important that these materials be utilized in spacecraft structures. One application where composites are being utilized with great success is for nozzle materials. As more mechanical and physical data become available, composite materials should become candidates for spacecraft structural components. With this in mind, the Materials and Processes Laboratory at Marshall Space Flight Center undertook a task to bring together the expertise and facilities available within the laboratory to process and characterize polymer composite materials. This task entailed producing and processing a characterization flow diagram and then testing the diagram by producing composite test specimens and, subsequently, characterizing and mechanically testing the material for damage tolerance properties. An example flow diagram is shown in the appendix.

II. MATERIALS AND SPECIMEN PREPARATION

The material selected was IM6/3501-6 carbon/epoxy manufactured by Hercules, Inc. This material was chosen since it was identified as one of the leading candidate materials for the space station rack structure. This material is one of the "older generation" fiber/resin systems and does not possess the strength and damage tolerance of the "new generation" fiber/resin systems. It came supplied in a unidirectional prepreg form on a 137-cm wide spool. Four types of specimens were to be prepared. These specimens were to be used for instrumented impact, compression, tensile and compression-after-impact (CAI) testing.

The tensile test specimens were fabricated as 8-ply quasi-isotropic layups with a (0,+45,-45,90)_s configuration. The impact, compression, and CAI specimens had the same configuration but were 16 plies in thickness. The cure cycle used was that recommended by the supplier; a 3 °F/min ramp to 350 °F with a dwell time of 2 h and a cool down at 5 °F/min all at a pressure of 551 kPa (80 psi). The actual cure cycles are given in the appendix. The tensile, compression, and CAI test specimens were fitted with fiberglass end tabs before machining. The tensile specimens were cut into 22.9- by 2.54-cm (9- by 1-in) coupons and the compression specimens were cut into 11.4- by 0.635-cm (4.5- by 0.25-in) coupons. The specimens to be impacted were machined into squares 11.1 cm (4.375 in) on a side. CAI specimens were 7.62-cm (3-in) wide and 17.8 cm (7 in) in length.

III. TEST METHODS

A. Nondestructive Evaluation (NDE)

The specimens (except CAI) were evaluated with ultrasonic C-scan before mechanical testing. After the plate specimens were impacted, even numbered specimens were examined with CT using ZnI penetrate enhancement. These specimens were also x rayed using +15° and 0° beam incidence. The C-scan results of the postimpact tests are given in the appendix.

B. Mechanical Testing

Ten tensile coupons were tested for tensile strength using American Society for Testing Materials (ASTM) standard D3039. An Instron 1125 loading frame was utilized with a crosshead rate of 0.127 cm/min (0.05 in/min). No measurements of material modulus were attempted.

Nineteen compression coupons were tested using ASTM standard D3410 (Celanese Compression). The same loading frame and crosshead rate used for the tensile coupons were used for the compression tests.

Impact studies were performed with a Dynatup 8200 drop weight impact apparatus with a falling weight of 1.21 kg (2.66 lb). The impactor was an instrumented hemispherical tup of 1.27-cm (0.5-in) diameter. Data were taken with a Dynatup 730 data acquisition system. The impact energies utilized ranged from 0.98 J to 19.5 J (0.72 to 14.4 ft-lb). The specimens were pneumatically clamped in place by two aluminum plates with holes of 7.62 cm (3 in). Specimen damage was recorded visually in addition to the instrumented data provided by the 730 acquisition system. Selected panels were viewed and photographed in cross section using a stereomicroscope at magnifications ranging from $\times 10$ to $\times 64$. These photographs were compared to the post-impact NDE records to examine the correlation.

CAI tests were carried out with the use of a new CAI fixture that has been successfully used in other studies within the Materials and Processes Laboratory. Specimens were hit with impact energies ranging from 1.5 to 18.2 J (1.1 to 13.4 ft-lb). A crosshead displacement rate of 1.27 mm/min (0.05 in/min) was used to load the specimens to failure.

IV. TEST RESULTS

A. Tensile Testing

The average tensile strength was measured to be 615 MPa (89,249 psi) with a standard deviation of 36.4 MPa (5,278 psi). The complete data for these tests are given in the appendix.

B. Impact Testing

Visible damage was not evident in samples until energy levels of 6.1 J (4.5 ft-lb) were achieved at which point a small dent could be felt on the impacted surface, and a hairline crack parallel to the outer fibers was visible on the back (nonimpacted) surface. Fiber breakage in the

specimens was not observed until 18.3 J (13.5 ft-lb) of impact energy was used. The instrumented output data were typical of all other carbon/epoxy systems tested. The force-time curve was relatively smooth until fiber breakage occurred, at which point a sharp drop in force was seen at the peak force level. Instrumented impact outputs are given in the appendix for each energy level tested.

C. NDE Testing

The C-scan results show that the nonimpacted panels had good consolidation with no detectable debonds. After impact, the C-scan data did not detect damage until 2.0 J of impact energy was used. At this level, areas of delamination show up as circular white areas directly under the point of impact. As the impact energy increased, the size of the white circle showing on the C-scans grew proportionately. At impact energies of 12 J or more, the white circles become more oblong in shape, indicating delaminations occurred to a greater extent between certain plies.

The x-ray data could better show in which layer the delaminations were most severe. Fiber orientation was very easy to see on the x rays, thus giving more detailed information about the specimen's damage state.

D. Cross-Sectional Observations

Cross-sectional photographs of the damage zone in each impact energy level used are given in the appendix. The specimens were unusually thick at 3.05 mm (0.16 in). Comparisons with other materials showed that each layer of the IM6/3501-6 was thicker than most other carbon/epoxy systems made from prepreg material.

Damage was not detected until the 3.1-J (2.26-ft-lb) energy level was used, at which point very small delaminations could be seen between the 10th and 11th plies from the top and between the 14th and 15th plies. At a slightly higher impact energy level, 3.99 J (2.91 ft-lb), a larger delamination can be seen, especially between the bottom two layers. Cracks running between plies is also evident at this energy level. The delaminations become no more severe until the 7.0-J (5.18-ft-lb) energy level is reached. At this impact energy level, a large crack can be witnessed running between the center plies. At the next energy level used, 12.1 J (8.91 ft-lb), severe delaminations are seen between almost every ply. Matrix cracks in each ply parallel to the fiber direction in that ply are very prevalent, especially in the bottom layers. At 12.5 J (9.26 ft-lb), fiber breakage can be seen in the 90° plies, and at larger impact energy levels, massive damage is witnessed.

E. CAI Testing

A total of seven specimens, impacted at seven different impact energies, were tested for residual compression strength. The smallest impact energy used, 1.5 J (1.1 ft-lb), caused a drop in strength of 25 percent over the undamaged strength, even though no visible cross-sectional damage is present at this impact energy. This fact points out the brittleness of the 3501-6 resin system since the failure initiation site could not be seen (at $\times 64$) at this impact energy. In addition, it should be noted that the 25-percent drop in compressive strength occurred at an impact level below that detectable by the NDE methods. At the point where internal damage was detected, 3.1 J (2.26 ft-lb), a drop in strength of approximately 39 percent from the undamaged strength occurred. The next energy level used in the test, 7.5 J (5.5 ft-lb), caused a drop in

strength of about 54 percent. Further increases in impact energy did not cause a further drop in residual strength, as shown by the residual strength versus impact energy plot given in the appendix.

F. Data Recording

All data pertaining to the material being evaluated are put in a notebook as a hard-copy record stored in EH33. Mechanical and physical data, as well as all photographs, are placed in sheet protectors which are then placed in a ring binder notebook for future reference.

V. OBSERVATIONS ON THE COMPOSITE IN-HOUSE FABRICATION, CHARACTERIZATION FLOW

The total time to process and characterize this material was approximately 10 months. A time line is given in the appendix showing how long each flow step required. It should be noted that this was a pilot effort to demonstrate an in-house Laboratory activity, and did not have high coordinated Laboratory priority. The panel processing was long due to the other priorities within the processing cycle. NDE analysis was prolonged due to equipment failure, which has since been corrected. It is anticipated that the total processing/characterization cycle could readily be reduced to 1 to 2 months for flat panel configurations with appropriate priority. If processing priorities cannot be resolved for future composite tasks, Polymers and Composites Branch has the capability to fabricate 12- by 12-in flat panels at a rate of 12 per day.

The area of machining the various composite specimens to precise dimensions represents a significant impact to the overall flow of this activity, primarily due to problems with generation of carbon dust and limited facilities to adequately accommodate the dust. The resources available to Materials and Processes Laboratory for machining this type material have not proven satisfactory for producing large numbers of precisely dimensioned specimens in a reasonable timeframe. Alternate approaches to this part of the fabrication flow are being assessed by Polymers and Composites Branch.

VI. CONCLUSIONS

The study demonstrated the feasibility of performing a complete composite processing/characterization flow utilizing the resources within the Materials and Processes Laboratory.

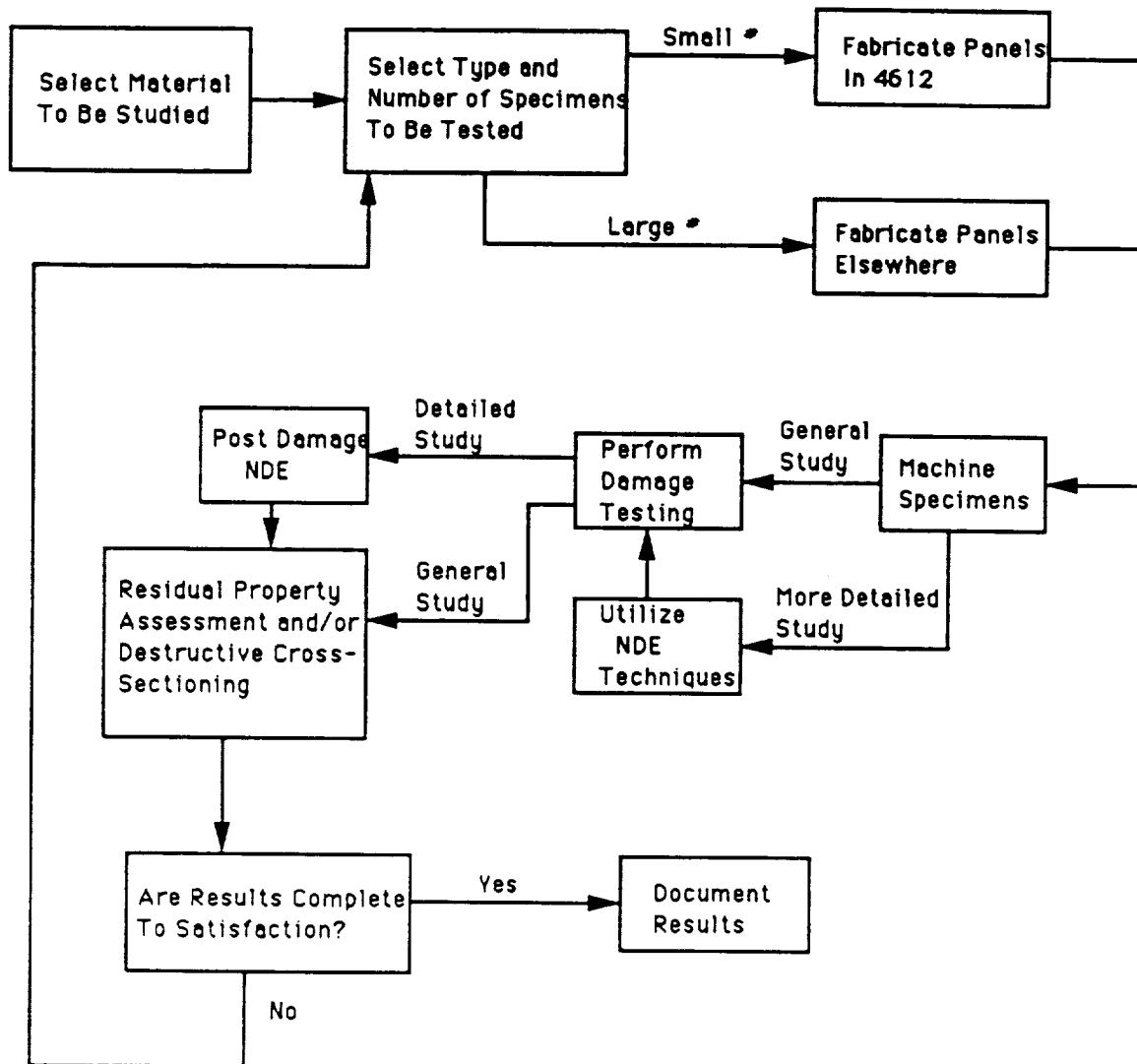
It is evident from this study that the composite processing/characterization time line should and can be compressed significantly to provide a reasonable response time for generation of material data bases.

Damage tolerance data on IM6/3501-6 carbon/epoxy provided a reasonable comparison to the prior data base for this material. The CAI data specifically demonstrated how critical impact damage can be for this older generation of untoughened materials.

The NDE results showed damage occurring at a lower impact energy than could be detected by cross-sectional photomicrographs. However, the NDE analysis did not detect damage at the lowest impact level used in this study. Damage at this level was detectable only through CAI tests which demonstrated a 25-percent drop in compressive strength.

APPENDIX

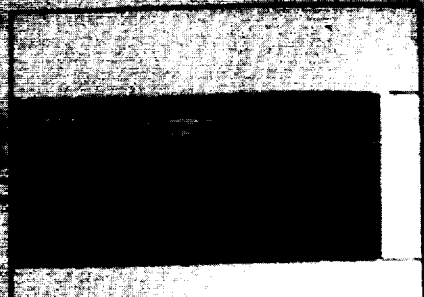
FLOW CHART FOR COMPOSITE MATERIALS DAMAGE TOLERANCE STUDIES



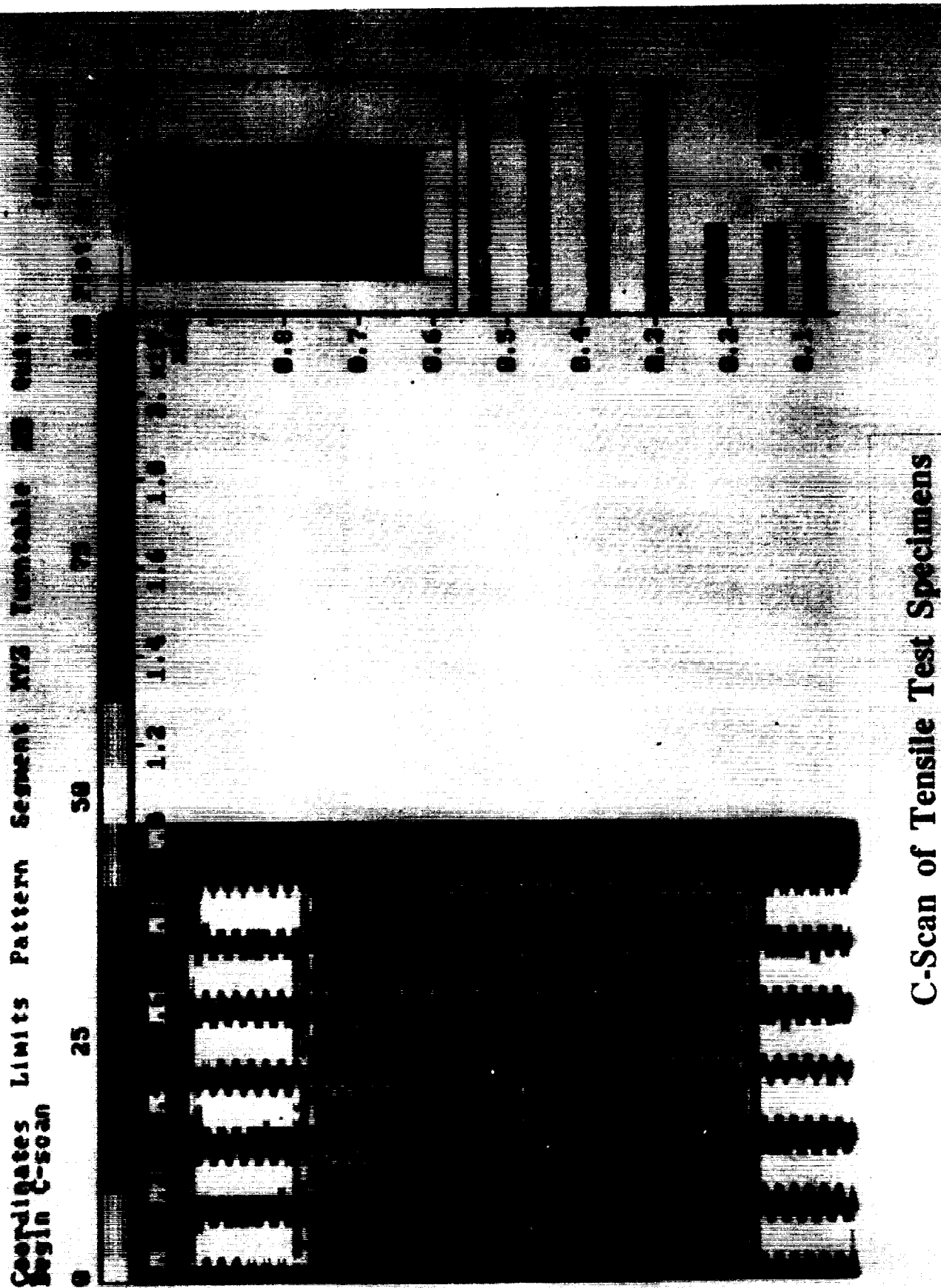
1. Use T300/934 Material and Try a Sample Run of the Above Process
2. Identify Problem Areas That Need to be Adressed
3. Work to Reduce Time Needed to Test a Material

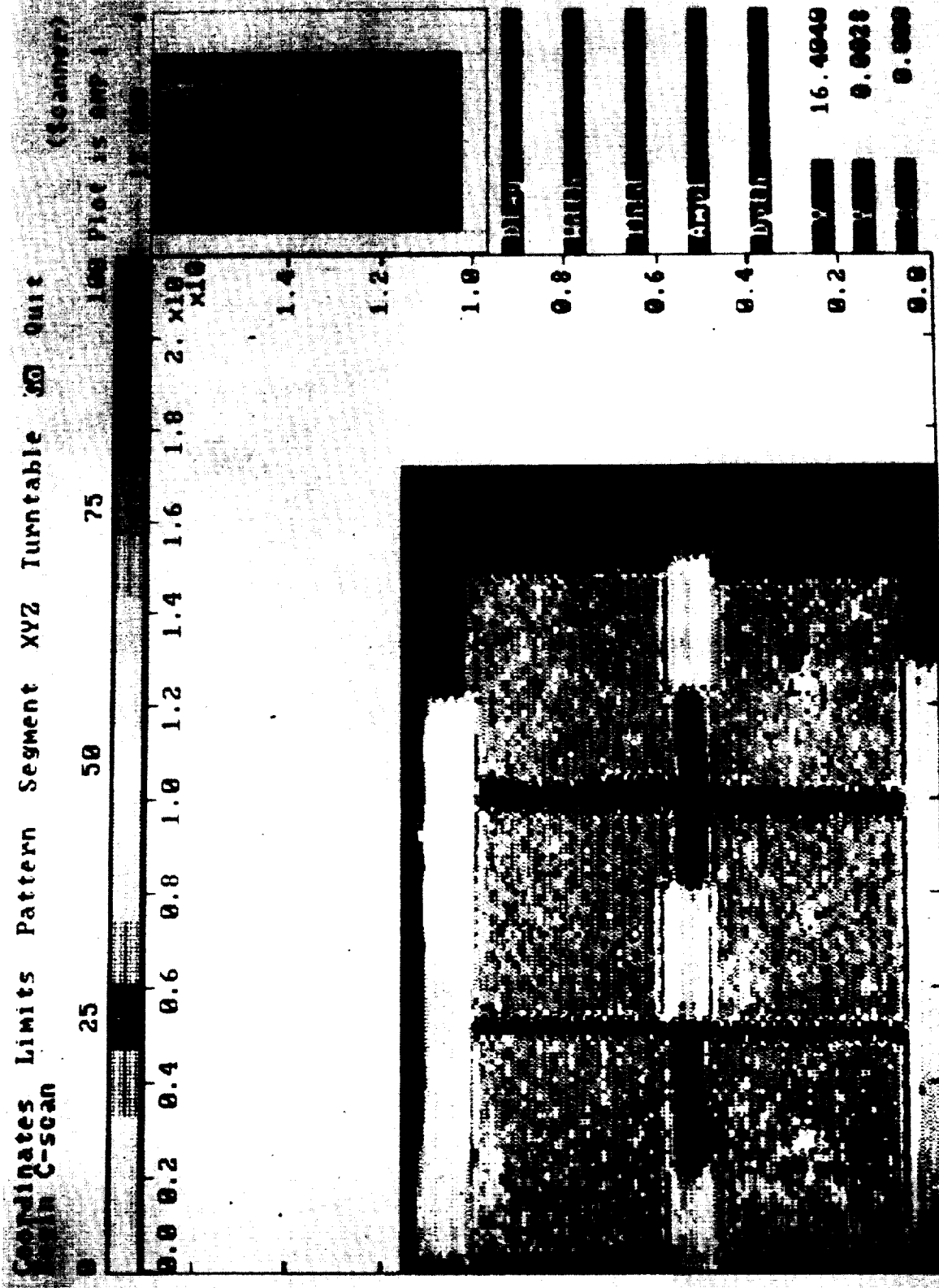
Coordinates Limits Pattern Segment XYZ Turntable XZ Units

25 50 75 100 Plot is 100-1

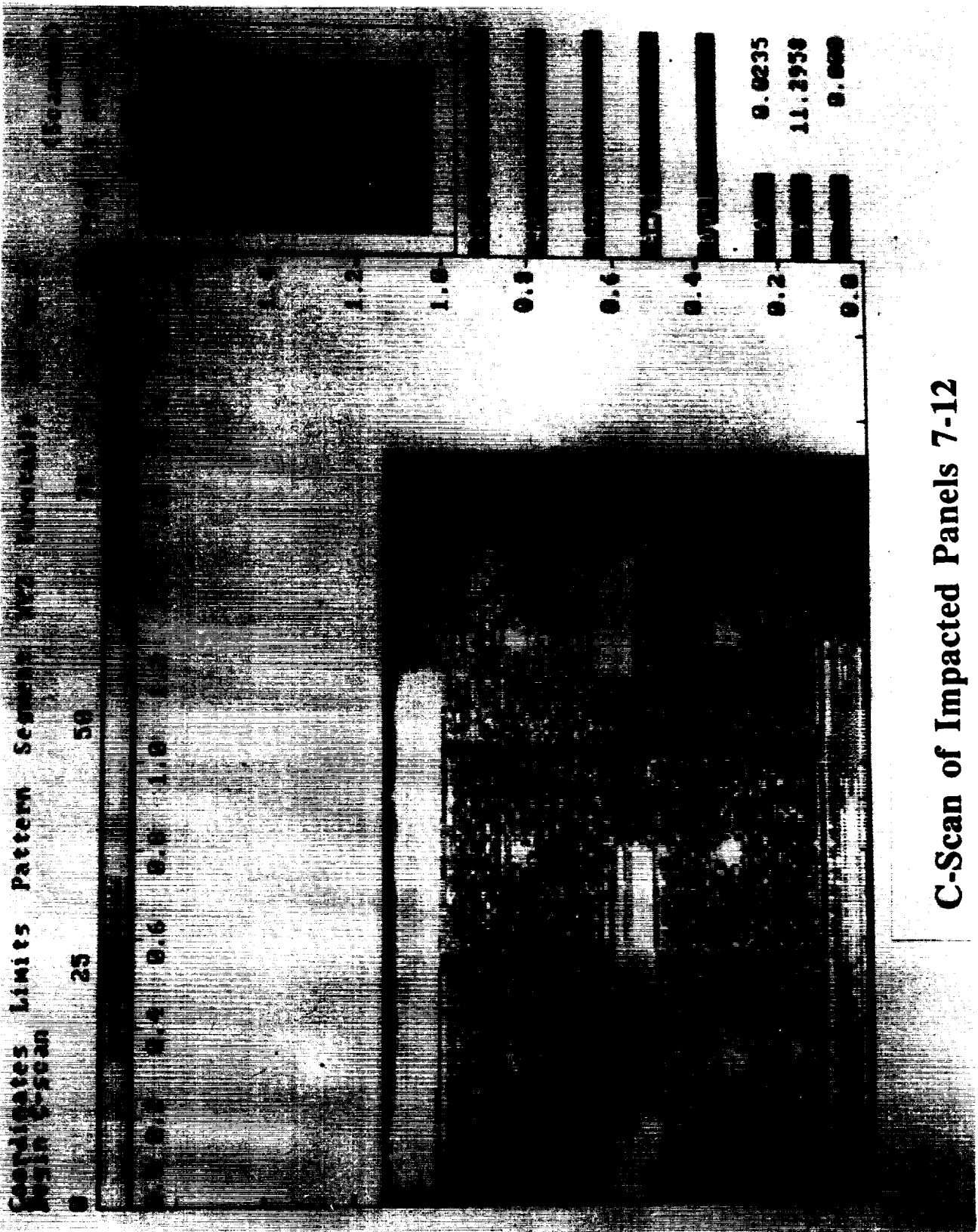


C-Scan of Tensile Test Specimens

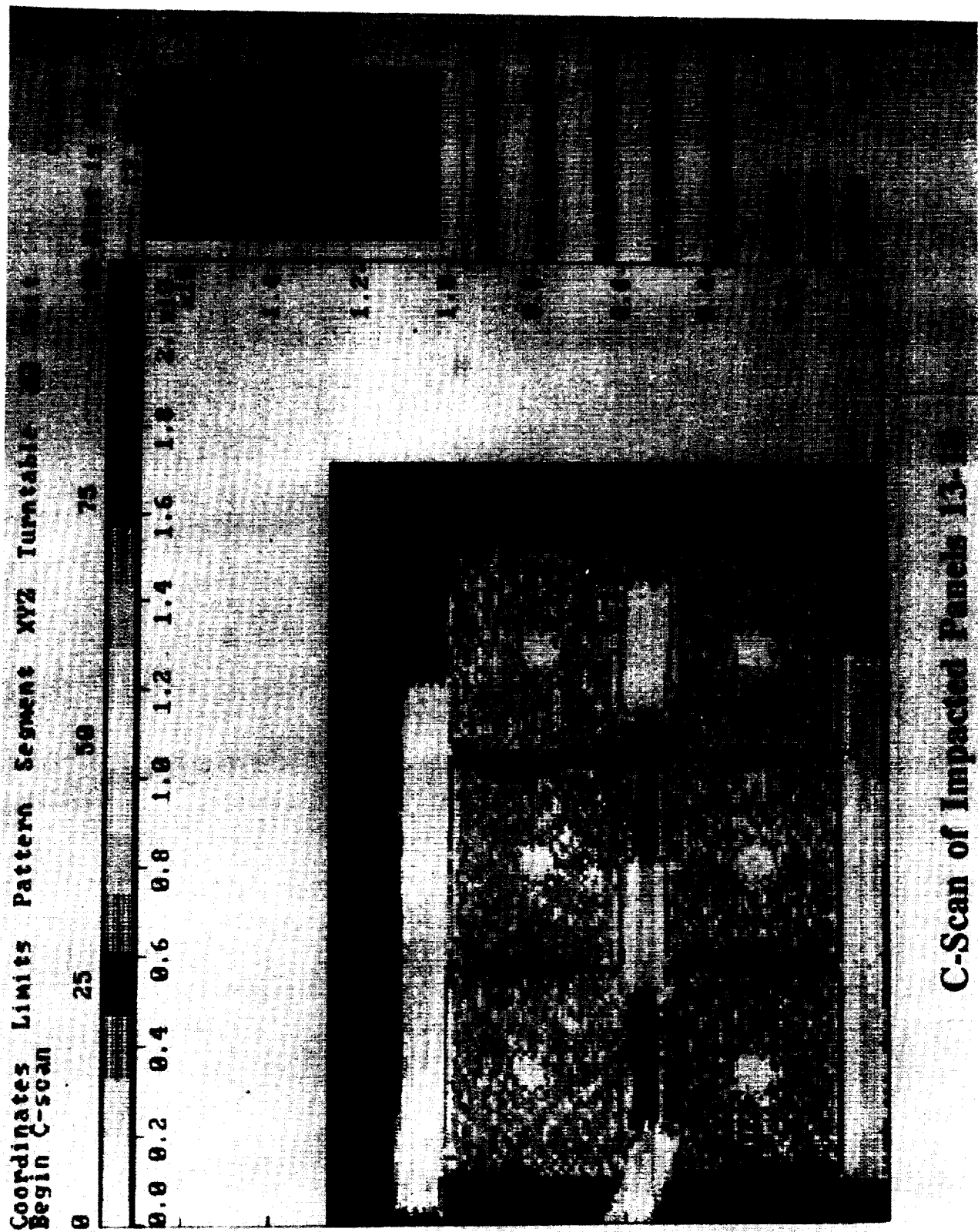




C-Scan of Impacted Panels 1-6

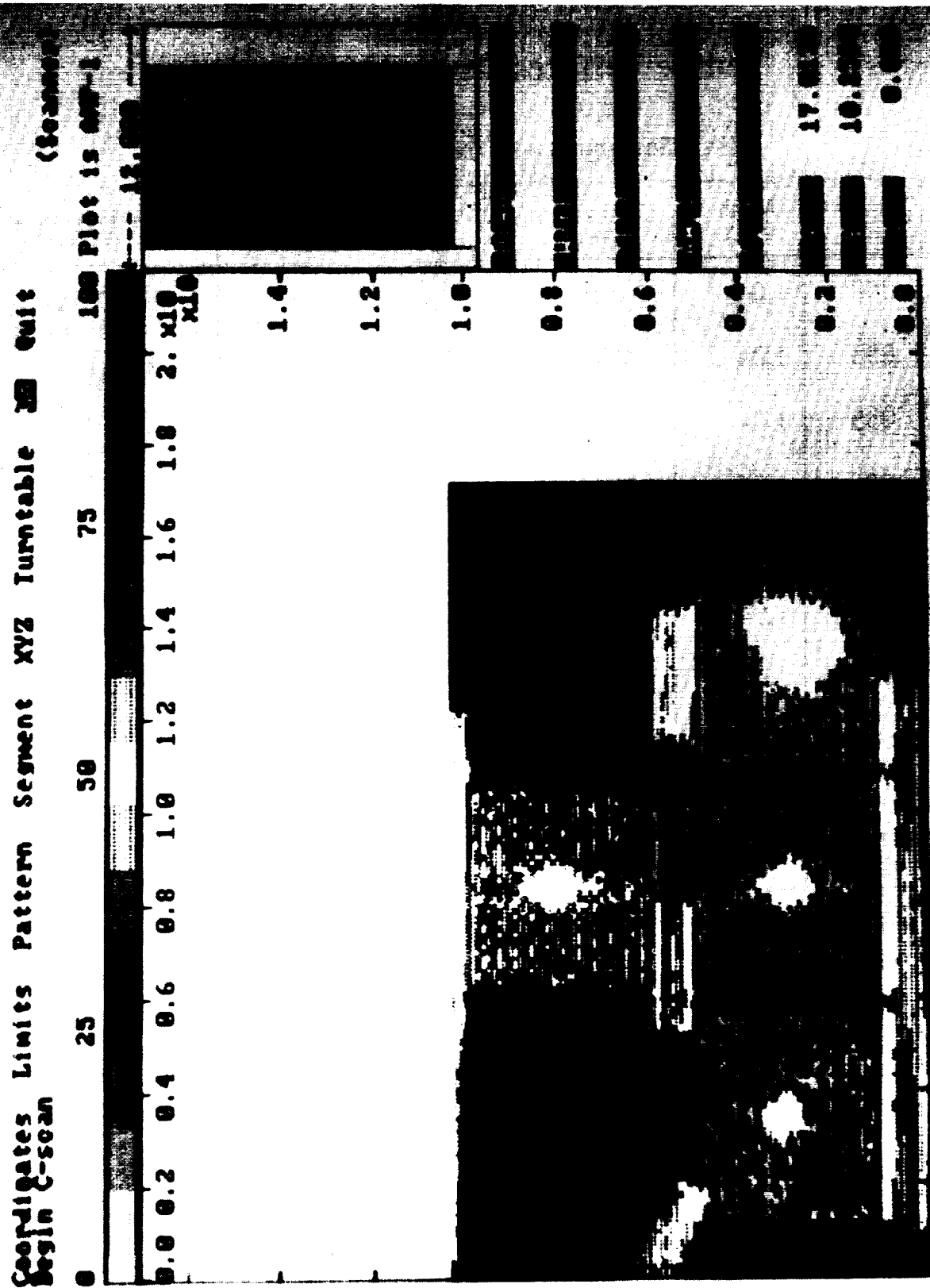


C-Scan of Impacted Panels 7-12





C-Scan of Impacted Panels 19-24

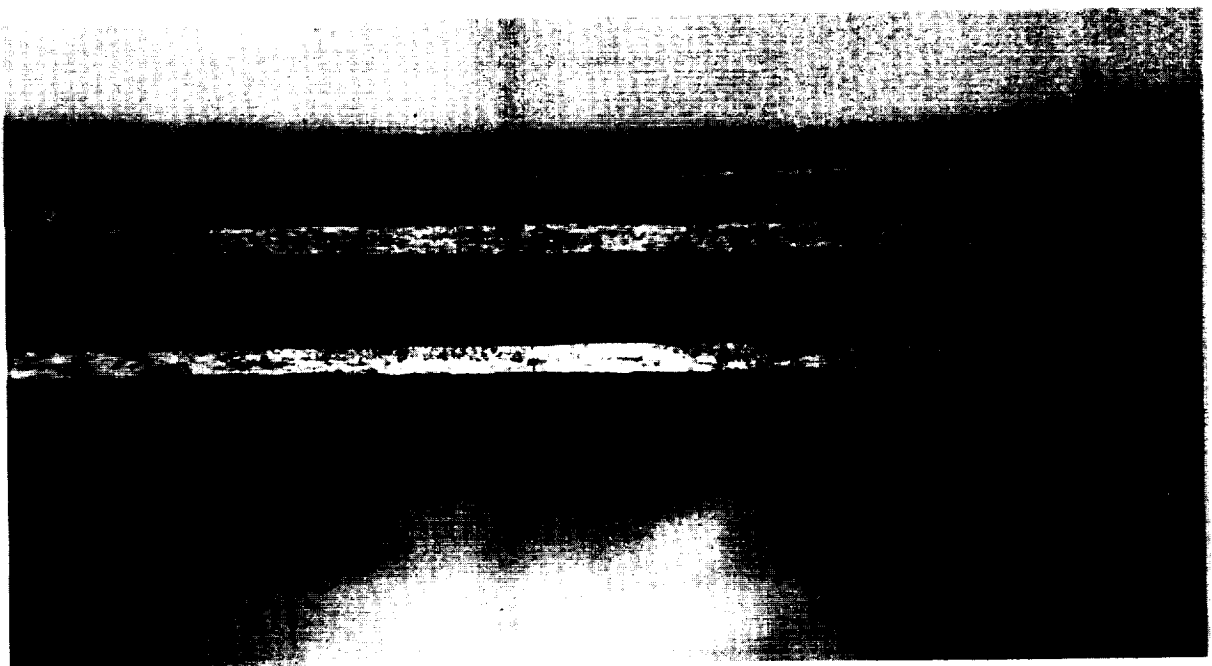
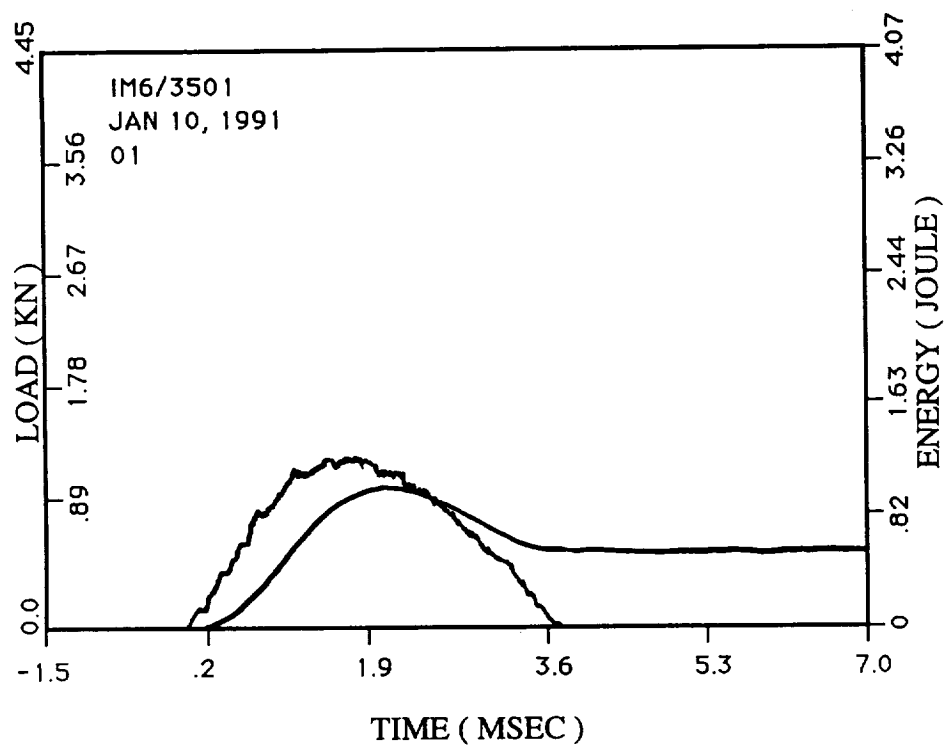


C-Scan of Impacted Panels 25-28

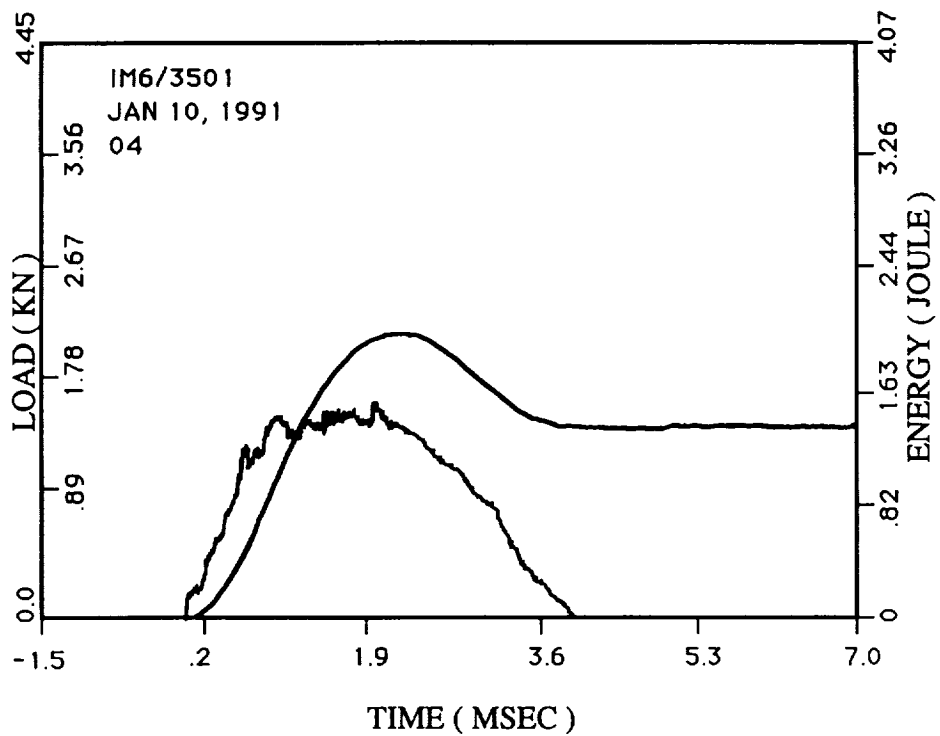
Tensile Test Results of IM6/3501-6

(Strain rate used = .05 in/min)

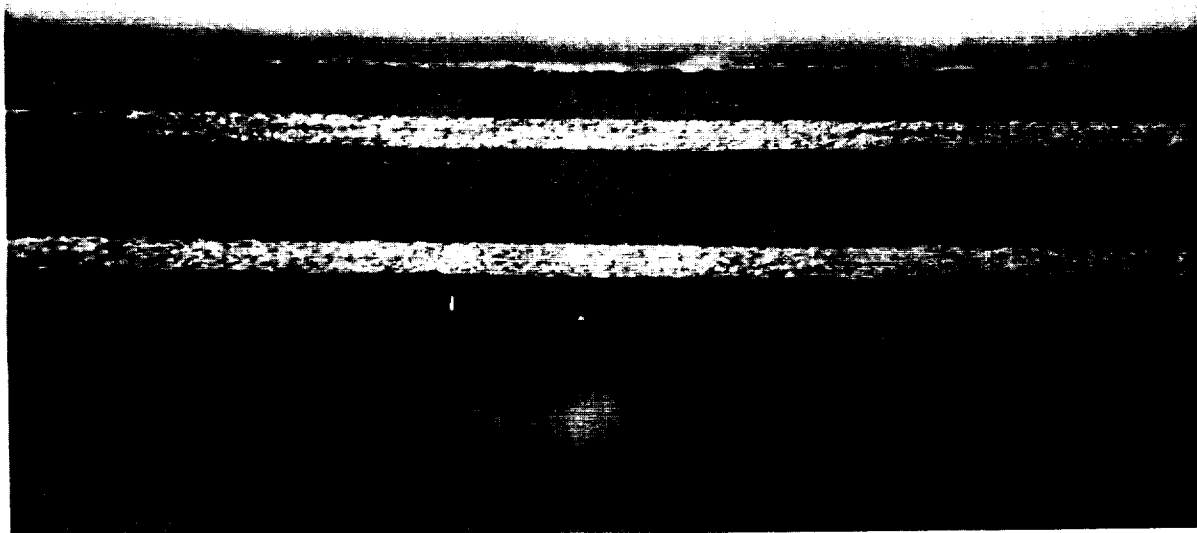
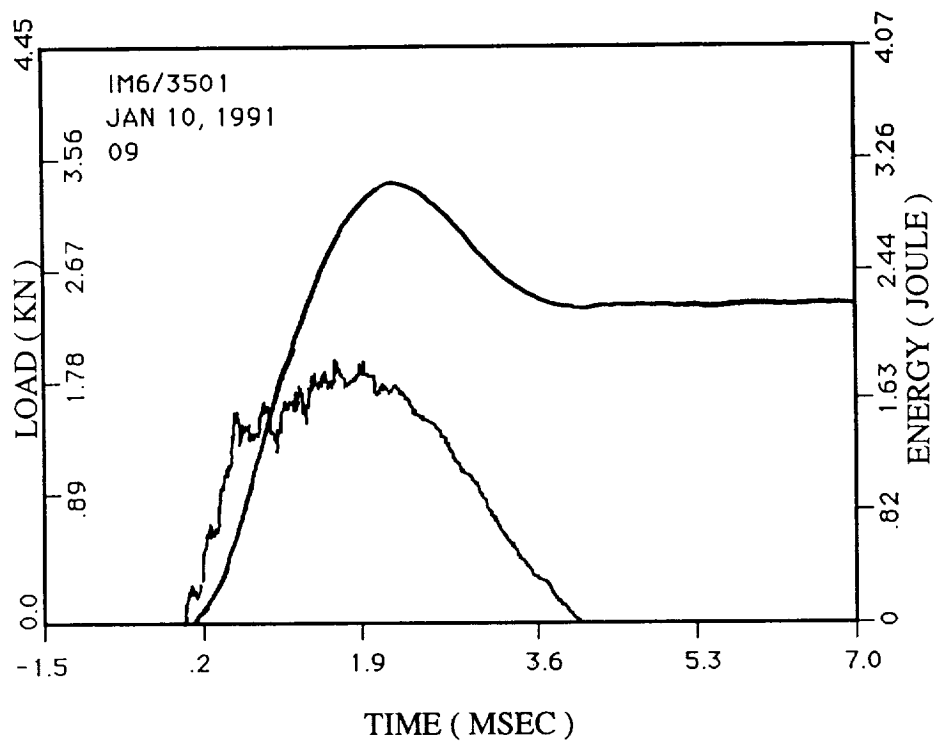
Specimen #	Thickness (in.)	Width (in.)	Load to Break (lbs)	Breaking Stress (PSI)	Breaking Stress (MPa)
C12	.058	1.04	5125	84964	585
C7	.058	1.04	4875	80819	557
C1	.055	1.04	5710	99825	688
C11	.055	1.04	5080	88811	612
C10	.056	1.04	5200	89286	615
C2	.057	1.04	5200	87719	604
C5	.056	1.04	5560	95467	658
C4	.055	1.04	4990	87238	601
C6	.057	1.05	5425	90643	625
C8	.057	1.05	5250	87719	604



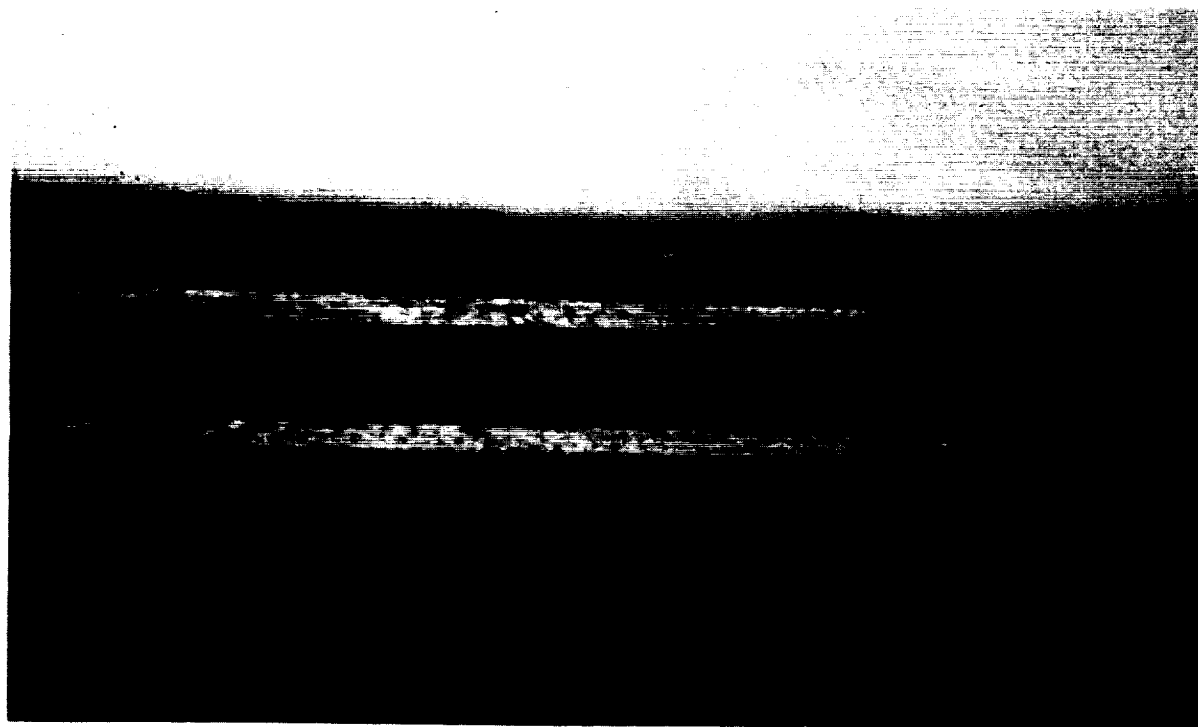
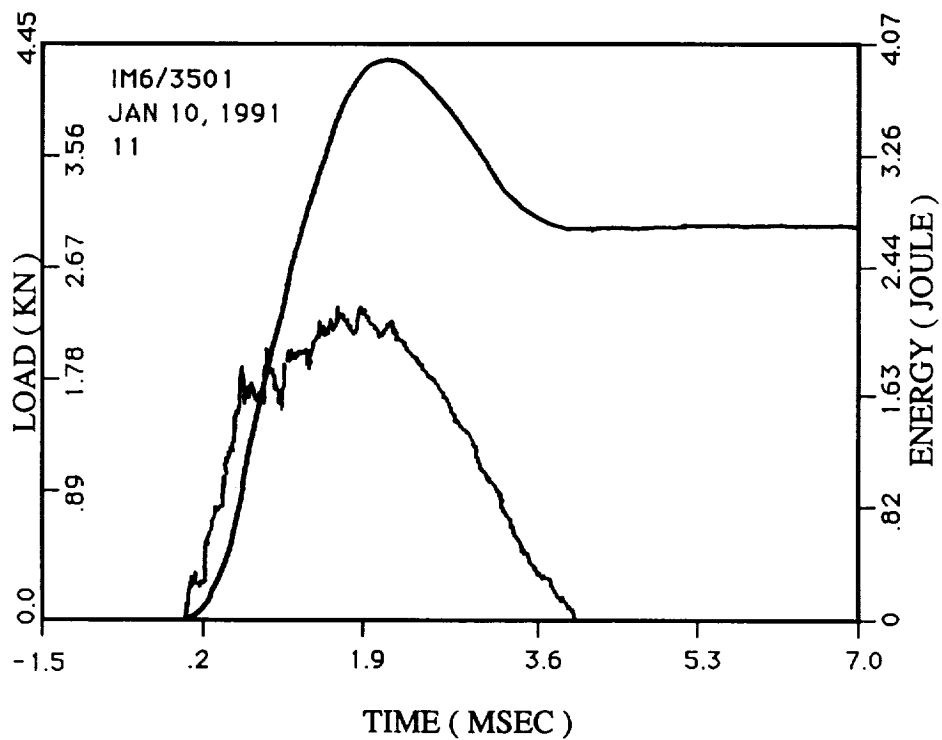
Force-Absorbed Energy-Time Plot and Cross-Sectional Photograph
of IM6/3501-6 Impacted at 0.98 J (0.72 ft-lb).



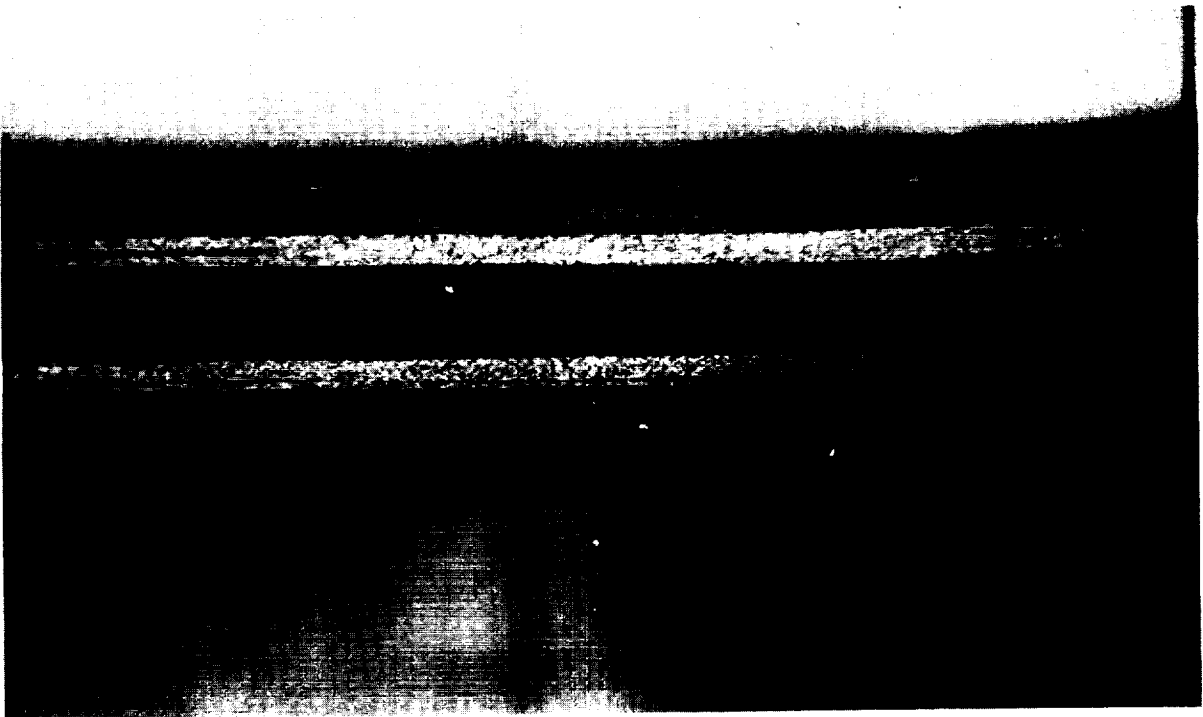
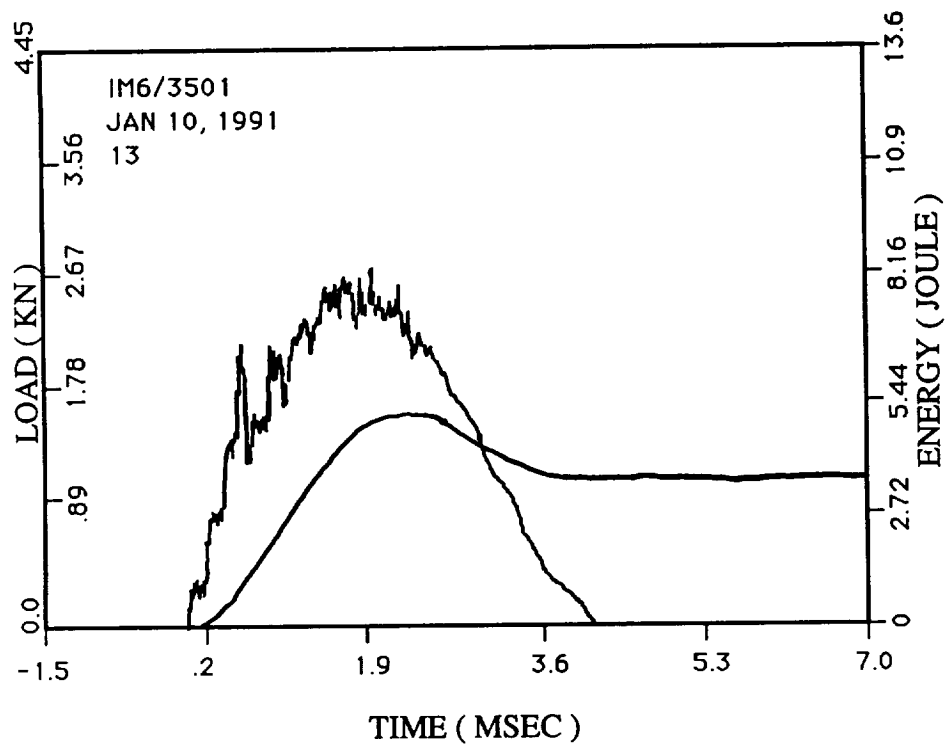
Force-Absorbed Energy-Time Plot and Cross-Sectional Photograph
of IM6/3501-6 Impacted at 2.0 J (1.5 ft-lb).



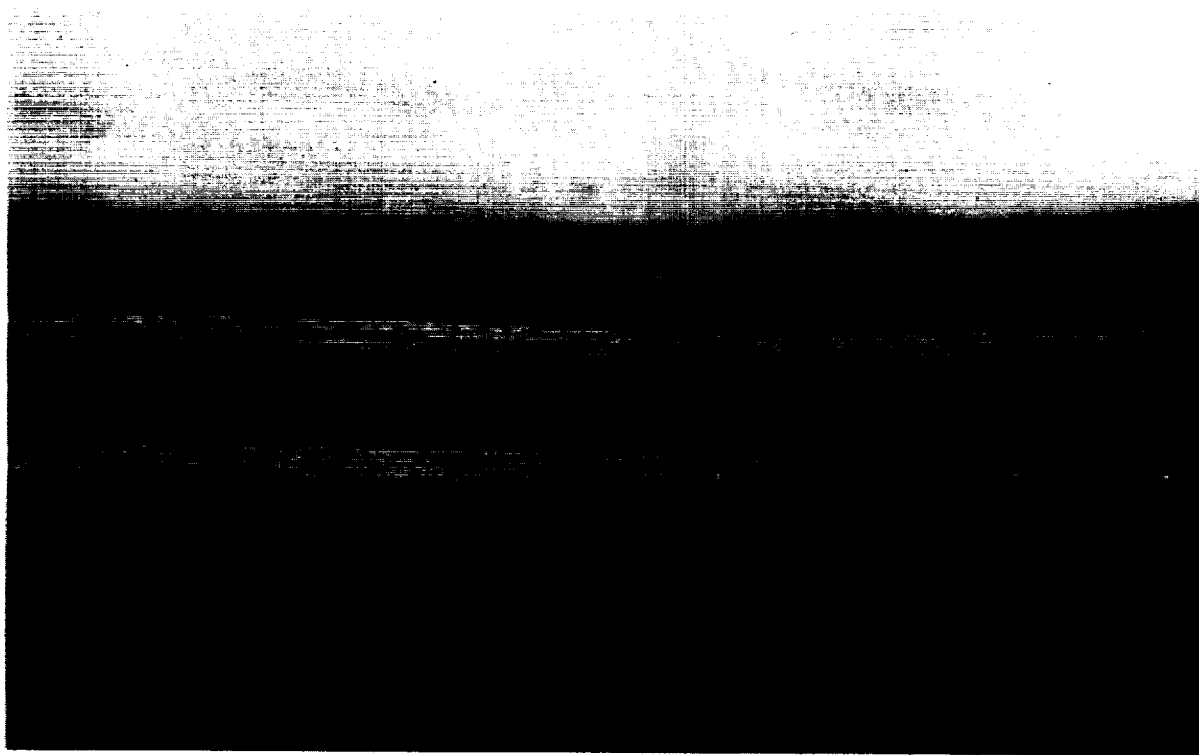
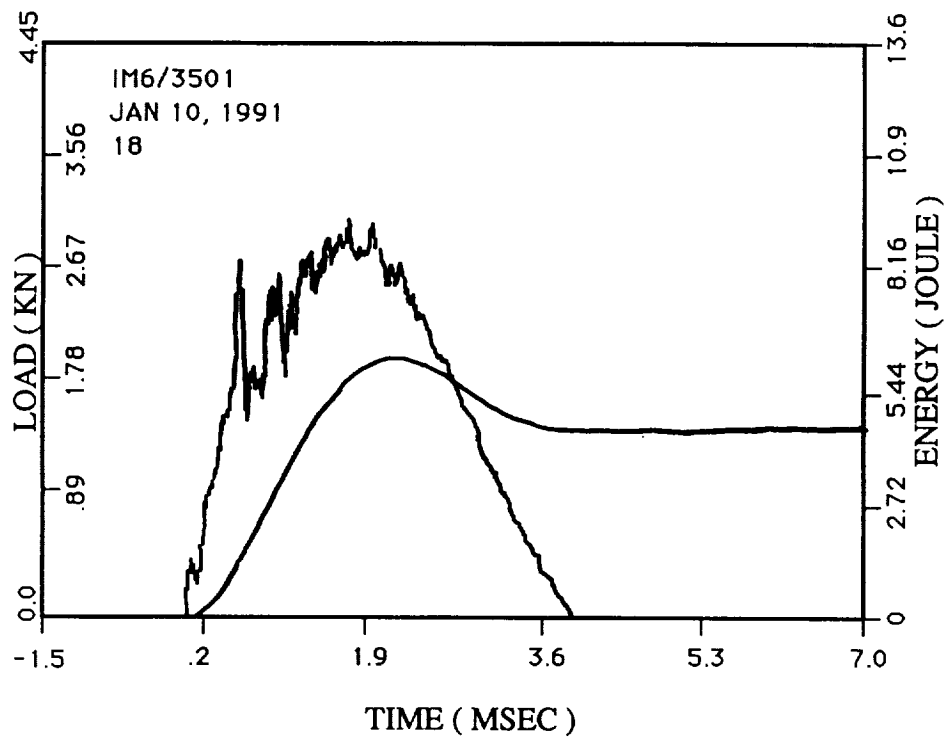
Force-Absorbed Energy-Time Plot and Cross-Sectional Photograph
of IM6/3501-6 Impacted at 3.1 J (2.3 ft-lb).



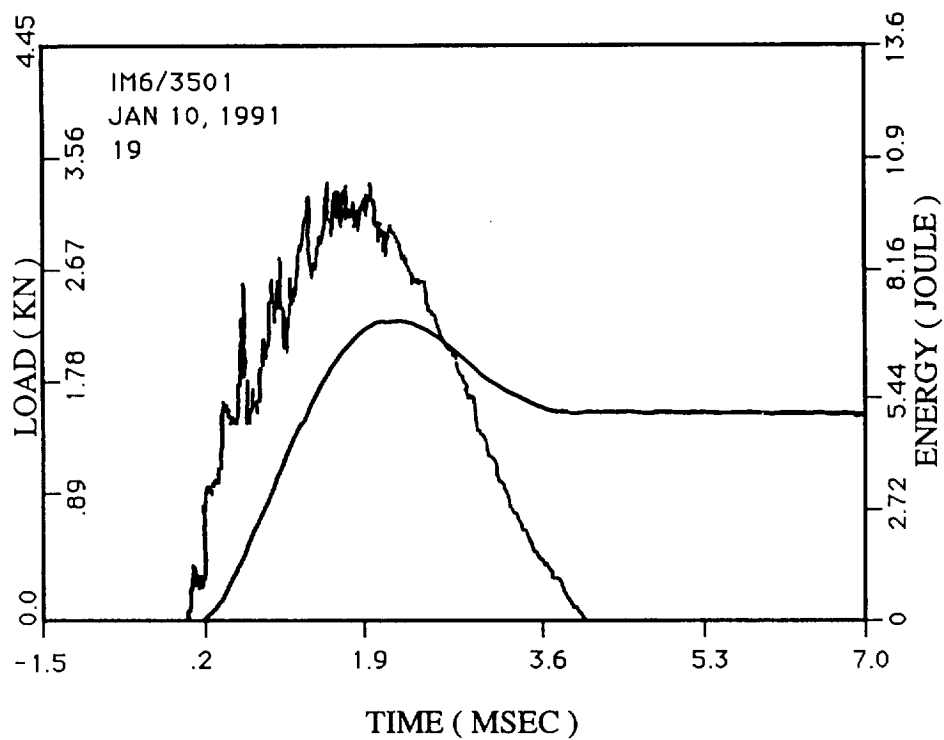
Force-Absorbed Energy-Time Plot and Cross-Sectional Photograph
of IM6/3501-6 Impacted at 3.9 J (2.9 ft-lb).



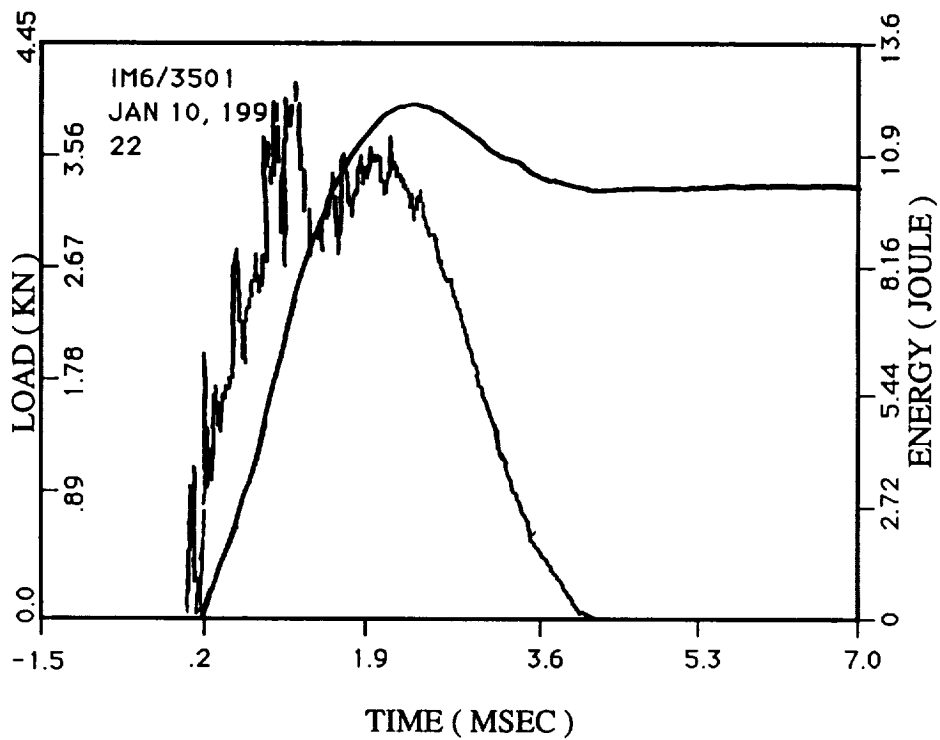
Force-Absorbed Energy-Time Plot and Cross-Sectional Photograph
of IM6/3501-6 Impacted at 5.0 J (3.7 ft-lb).



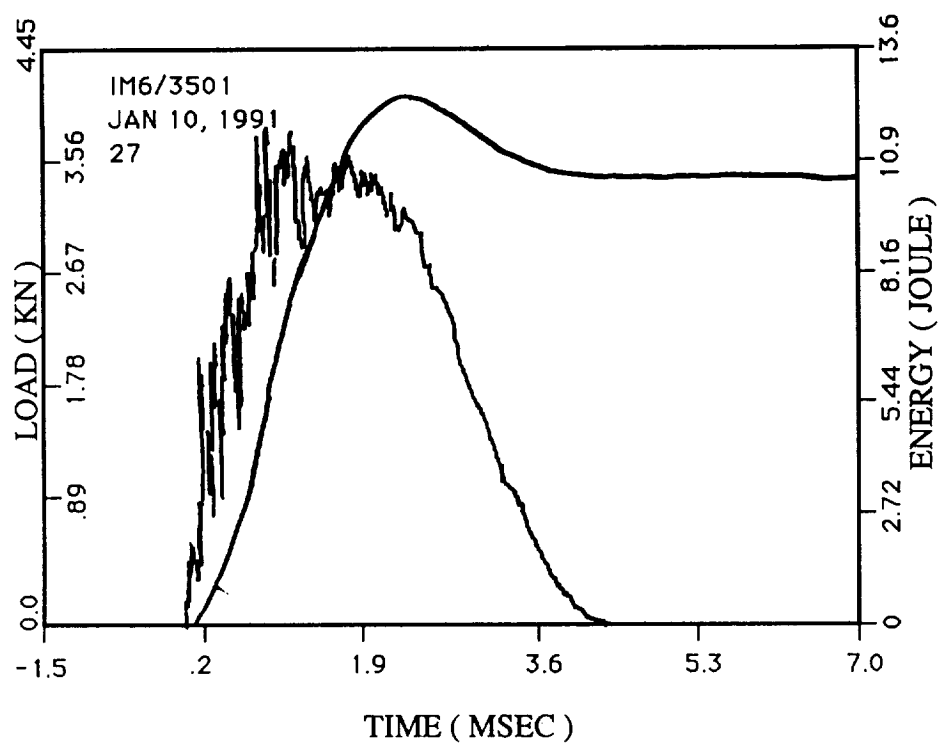
Force-Absorbed Energy-Time Plot and Cross-Sectional Photograph
of IM6/3501-6 Impacted at 6.1 J (4.5 ft-lb).



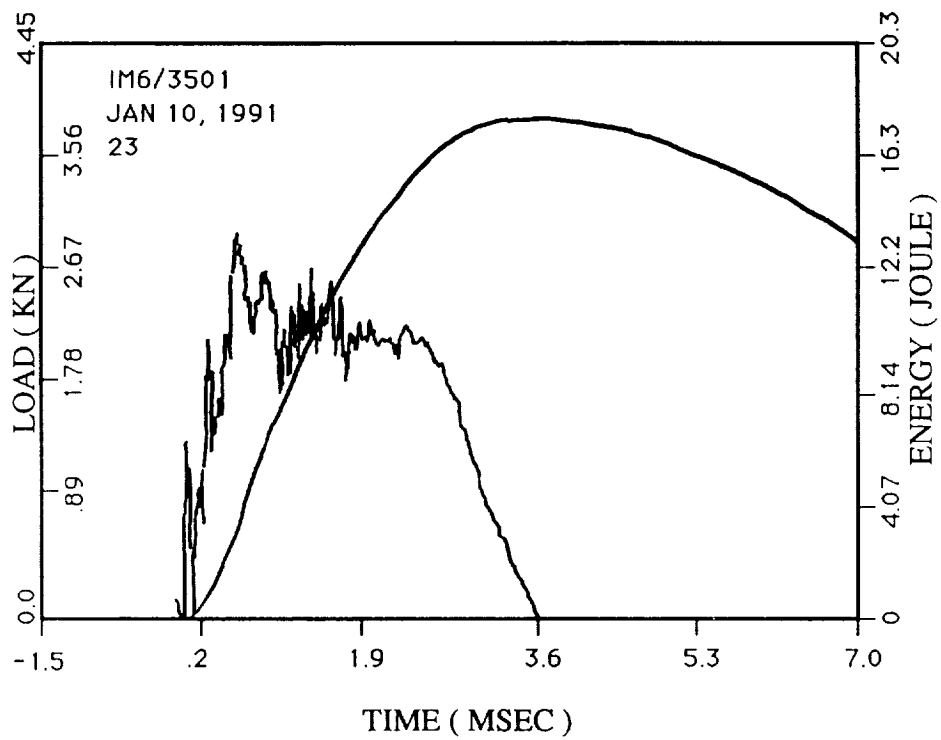
Force-Absorbed Energy-Time Plot and Cross-Sectional Photograph
of IM6/3501-6 Impacted at 7.0 J (5.2 ft-lb).



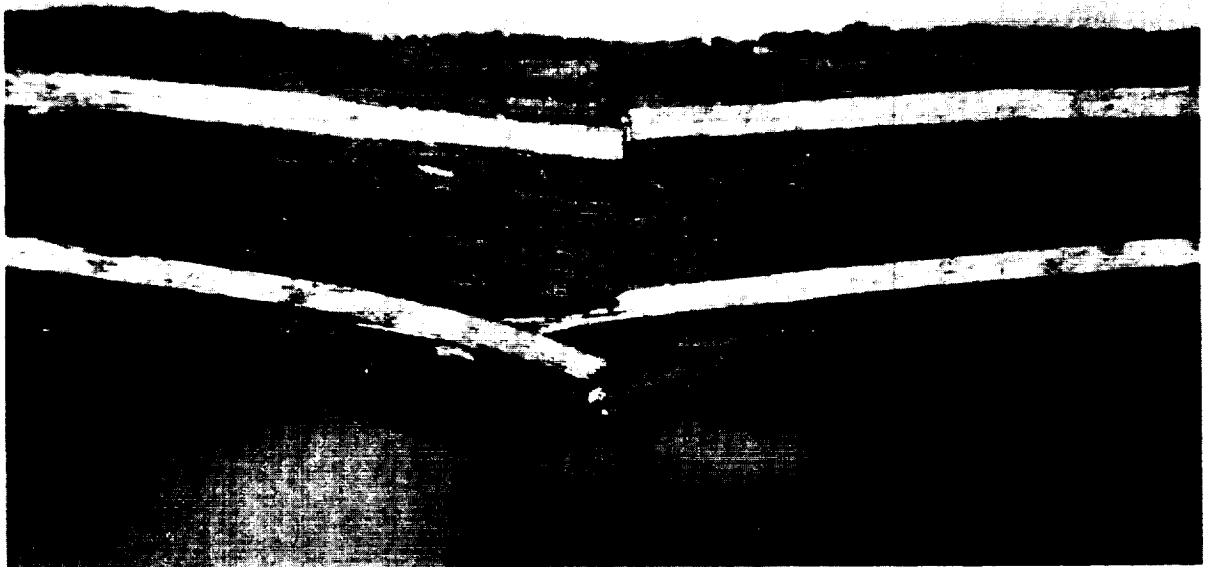
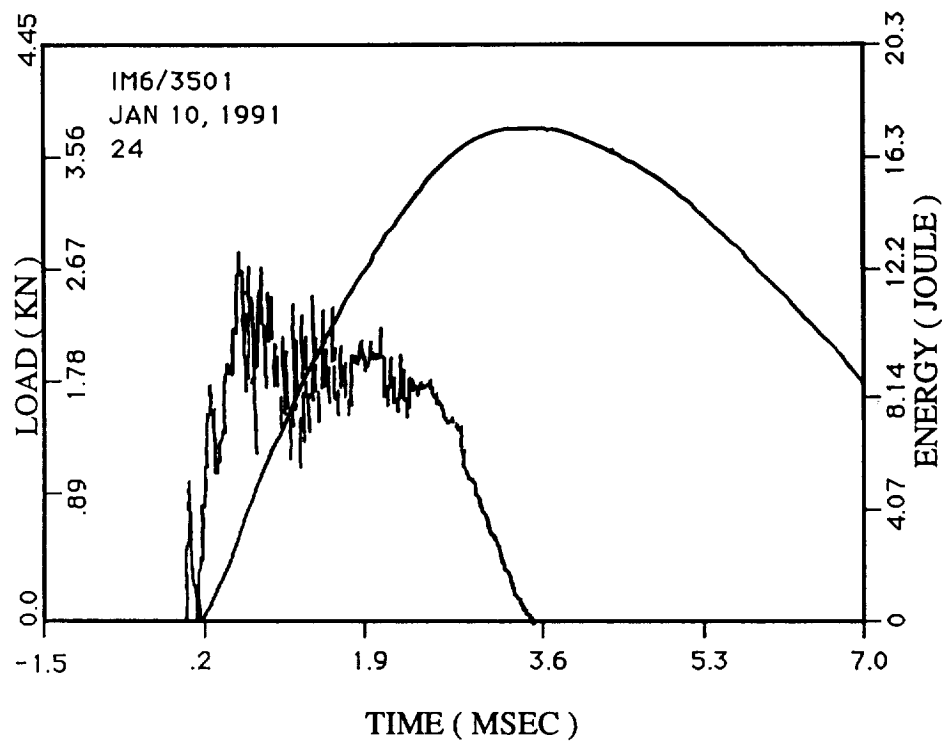
Force-Absorbed Energy-Time Plot and Cross-Sectional Photograph
of IM6/3501-6 Impacted at 12.1 J (8.91 ft-lb).



Force-Absorbed Energy-Time Plot and Cross-Sectional Photograph
of IM6/3501-6 Impacted at 12.5 J (9.20 ft-lb).



Force-Absorbed Energy-Time Plot and Cross-Sectional Photograph
of IM6/3501-6 Impacted at 18.3 J (13.5 ft-lb).



Force-Absorbed Energy-Time Plot and Cross-Sectional Photograph
of IM6/3501-6 Impacted at 19.5 J (14.3 ft-lb).

Task	1990							1991					
	May	June	July	Aug.	Sept.	Oct.	Nov.	Dec.	Jan.	Feb	Mar.	Apr.	May
Processing and Machining of Specimens (EH 41)													
NDE Examination, Undamaged Specimens (C-Scan & X-ray) (EH15)													
Specimens Impacted (EH33)													
Tensile Testing (EH33)													
C-scan and X-ray of Damaged Specimens (EH15)													
Compression Testing of Undamaged Material (EH33)													
Cross-Sectioning and Photography (EH33)													
Processing and Machining of CAI Specimens (EH33)													
CAI Testing (EH33)													

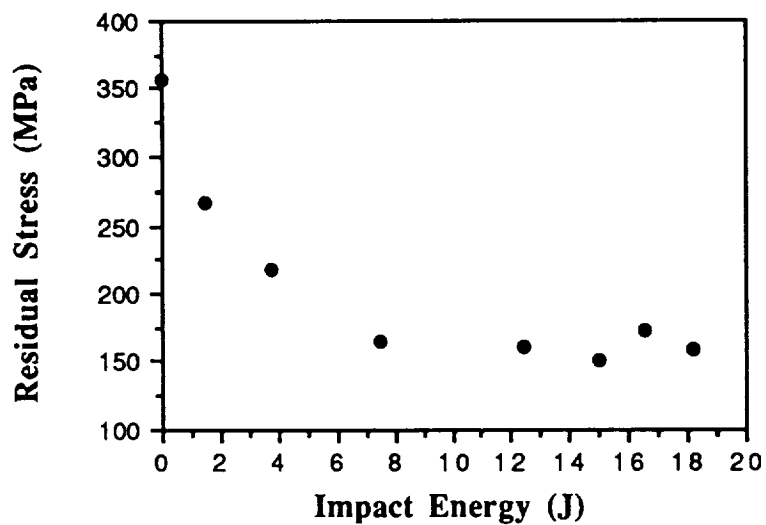
Time Line for Various Tasks

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MATERIALS CHARACTERIZATION TASK, PART 1. D... A. T. Nettles et al
(NASA) Sep. 1991 31 p
NAS 1.15:103553

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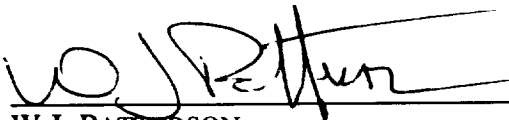
Residual Compression Strength versus Impact Energy for
IM6/3501-6

APPROVAL


MATERIALS AND PROCESSES LABORATORY COMPOSITE MATERIALS CHARACTERIZATION TASK, PART I. DAMAGE TOLERANCE

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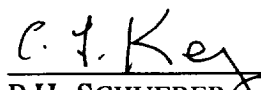
The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.



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Chief, Nonmetallic Materials Division



P.H. SCHUERER
Director, Materials and Processes Laboratory

